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TECHNICAL MEMORANDUM NO. 113

THE HEAT OF FORMATION  
OF  
TRISETHYLENEDIAMINE COBALT III PERCHLORATE  
(TEDCP)

CHARLES LENCHITZ  
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DECEMBER 1962



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Picatinny Arsenal Technical Memoranda No. 1113

THE HEAT OF FORMATION OF TRIETHYLENEDIAMINE  
COBALT III PERCHLORATE (TEDCP)

by

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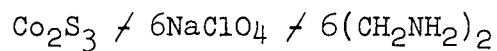
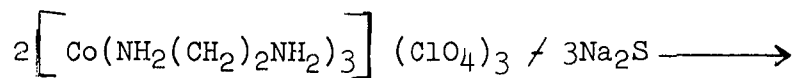
Feltman Research Laboratories  
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### ABSTRACT

The heat of formation ( $\Delta H_f$ ) of Trisethylenediamine Cobalt III Perchlorate (TEDCP) was calculated from the reaction:



and a value of -196 kcal/mole was obtained. The heat evolved from the above reaction was determined in a differential calorimeter.

## CONCLUSIONS

The determined heat of formation of TEDCP is -196 kcal/mole. The accuracy of this result is difficult to estimate, because of the uncertainties connected with this measurement.

## RECOMMENDATIONS

It is recommended that a different type of reaction be investigated for determining the heat of formation of Cobalt III salts.

## INTRODUCTION

The normal difficulties encountered in determining the heat of formation of organometallic compounds were adequately summarized by J. P. McCullough of the Bureau of Mines at the fourth meeting of the Thermochemical Panel (JANAF-ARPA-NASA in July 1961). In determining the  $\Delta H_f$  of TEDCP by combustion methods, one encounters two additional difficulties, explosive burning and the presence of halogens. The former further increases the probability that the metal (cobalt) will either not be completely oxidized or it will not be oxidized to any one uniform state. Because of this, it was decided not to use the rotating bomb calorimeter but to use reaction calorimetry for determining the heat of formation.

Circular 500 (Reference 1) lists the heat of formation of several cobalt complexes. An investigation of the source of this data shows that some of the values are based on the work of Lamb and Simmons in 1921 (Reference 2) and Ovenston and Terrey in 1936 (Reference 3). These investigators reacted complex salts with sodium sulfide and obtained a precipitate of  $\text{Co}_2\text{S}_3$ . The calculation of  $\Delta H_f$  is based however, on a calculated heat of formation of  $\text{Co}_2\text{S}_3$  solid. To correct to solid  $\text{Co}_2\text{S}_3$  in a solution of other salts or even in water is practically impossible, because  $\text{Co}_2\text{S}_3$  is easily oxidized and is extremely difficult to work with. The results obtained here as well as those reported previously must

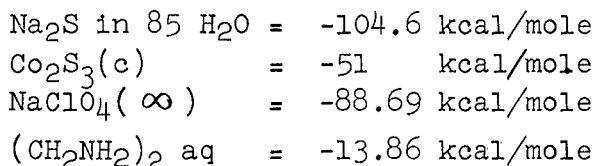


therefore eventually be corrected for this effect, or a different type of reaction investigated.

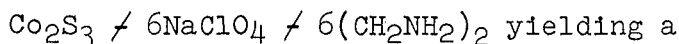
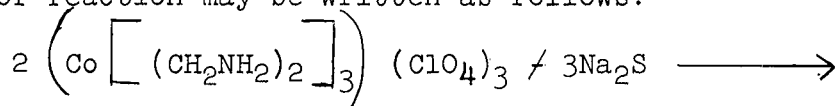
## RESULTS

The results obtained for the reaction between TEDCP and Na<sub>2</sub>S solution are listed in Table I. Columns (2) to (6) list the electrical calibration data. Column (7) shows the decrease in microvolts due to the reaction. Column (8) shows the heat of reaction in kcal/mole. A series of thirteen determinations were made yielding an average heat of reaction  $\Delta H_r$  of  $19.8 \pm 0.3$  kcal/mole (endothermic).

The following heats of formation, taken from Circular 500 (Reference 1) were used to calculate the heat of formation of TEDCP:



The heat of reaction may be written as follows:



$$\Delta H_f = -196 \text{ kcal/mole.}$$

## EXPERIMENTAL PROCEDURE

The heat of reaction was determined in a differential solution calorimeter. The calorimeter consists of twin 500cc Dewars, with matched manganin heaters, stirrers, breaking mechanism and a 30 junction 36 gauge differential copper constantan thermopile, all sealed to the lid of the calorimeter. The stirrers are driven by a common motor through an oil cup seal. The cover is bolted to the vessels and sealed with a teflon gasket. Thus the entire calorimeter is airtight.

To prevent attack by the  $\text{Na}_2\text{S}$  solution, the thermopile was placed in glass tubing (and immersed in oil). The stirrer was made of stainless steel, and the heater is also enclosed in stainless steel sheath.

All reactions were made between 26.3 and 26.6°C. The calorimeter being enclosed in a series of three wooden boxes with the outer box temperature controlled.

The calorimeter was conditioned overnight after which time equilibrium was reached and measurements made.

The TEDCP was loaded in glass ampoules. After determining that the hygroscopicity of the sample was nil, drying at high temperature was abandoned. Instead, the sample in its ampoule was placed in a vacuum dessicator which was placed in a dry box through which nitrogen was passed. The calorimeter was loaded and assembled in this nitrogen atmosphere as well as the  $\text{Na}_2\text{S}$  solution which was prepared in oxygen free water.

SAMPLE:

The sample was synthesized by the Universal Match Co. and recrystallized from water. The perchlorate content of the sample was then determined by precipitation with tetraphenylphosphonium chloride according to the procedure of Willard and Perkins (Reference 4). The average recovery of  $\text{ClO}_4$  is 99.64% of theoretical. The results of the analysis are listed below:

<u>Wt.</u> <u>Sample,</u> <u>mg.</u>	<u><math>\text{ClO}_4</math> Calc.,</u> <u>mg.</u>	<u><math>\text{ClO}_4</math> Found,</u> <u>mg.</u>	<u>Difference,</u> <u>mg.</u>
501.1	278.1	278.3	+ 0.2
503.9	279.6	278.8	- 0.8
500.5	277.8	276.0	- 1.8
501.6	278.4	276.7	- 1.7

Although the original work (Reference 4) reports errors only as large as 0.08 mg., these tests were made on  $\text{KClO}_4$  where chances of interference by other radicals and ions are negligible. Emission spectroscopy showed cobalt to be the only metal present. Analysis for cobalt by electrodeposition gave the following results:

Grams, Co,  
Actual

.0560  
.0568

Grams, Co,  
Calculated

.0553  
.0551

SODIUM SULFIDE:

"Fisher" Certified Reagent  $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$  with a Certificate of Analysis was used.

## DISCUSSION

The analysis of the products of reaction cannot be made on the calorimeter contents. This is due to the difficulty of handling the  $\text{Co}_2\text{S}_3$  precipitate. The precipitate is not only easily oxidized but is extremely difficult to transfer. Analogous experiments were therefore made in the dry box in a nitrogen atmosphere.

The perchlorate determinations made after the precipitation of the cobalt checked those obtained previously. These results are listed in Table II. The final recovery is 99.59% of the calculated value using the same method as previously described (Reference 4).

The analysis for  $\text{Co}_2\text{S}_3$  presents a problem. The procedure involves the recovery of sulfur from the precipitate as  $\text{BaSO}_4$ . In all of the ten determinations, there is found an excess of sulfur (Table III).

Assuming the precipitate to be  $\text{Co}_2\text{S}_3$ , analytical values up to 10% in excess of the anticipated values are obtained. An emission spectrographic analysis of the precipitate for sodium shows that only cobalt is present (Sodium sulfide would be the most likely occlusion in the precipitate, because the reaction was determined with an excess of  $\text{Na}_2\text{S}$ ).

The most plausible explanation for this behavior is that the  $\text{Co}_2\text{S}_3$  is further oxidized to  $\text{CoS}_2$ , and that a mixture of the two is probably present. It is also noted in the analysis for  $\text{Co}_2\text{S}_3$  made by Lamb and Simmons (Reference 2) a slight excess of sulfur is reported in each of their determinations.

An additional check on the purity of the compound (TEDCP) was made by burning the sample under 30 atmospheres of oxygen in a combustion bomb and analyzing for carbon dioxide. The two results obtained are 97.30% and 99.79% of the calculated value. Considering that the compound burns explosively, one can assume that the carbon content represents an essentially pure TEDCP. Each analysis for  $\text{ClO}_4$  confirms this purity.

Although the chance formation of  $\text{CoS}_2$  in the actual calorimetric process (which is sealed) is not as great as in the reaction made outside the calorimeter, it must be assumed that some  $\text{CoS}_2$  is formed along with  $\text{Co}_2\text{S}_3$ . The extent of formation of  $\text{CoS}_2$  may be a contributing factor to the

significantly larger standard deviation ( $\sigma$ ) of the heat of reaction ( $\Delta H_r$ ) experiments over the electrical calibrations. Thus  $\sigma$  for the calibration experiments range from 0.11% to 0.29% in comparison to the heat of reaction experiments where the average value is 19.8 kcal/mole and  $\sigma$  is 0.3 kcal/mole, which is equivalent to 1.5%.

The value for the heat of formation of TEDCP (-196 kcal/mole) must therefore be subject to this correction as well as the other correction mentioned previously, i.e.  $\Delta H_f \text{ Co}_2\text{S}_3$  in solution.

Because of these errors, temperature correction to 25°C. is considered to be insignificant as well as corrections for the vapor phase above the calorimeter.

#### REFERENCES

1. Rossini, F. D., Wagman, D. P., Evans, W. H., Levine, S., Jaffe, I.; Circular 500, "Selected Values of Chemical Thermodynamic Properties", National Bureau of Standards, February 1952.
2. Lamb, A. B., Simmons, J. P.; "The Heats of Solution and of Transformation of the Acido and Aquo Cobalt Pentammines", J. Am. Chem. Soc. 43, 2188 (1921).
3. Overston, T. C. J., Terry, H.; "Heats of Formation and Solution of Some Isomeric Cobaltammines", J. Chem. Soc. 1660 (1936).
4. Willard, H. H., Perkins, L. K.; J. Anal. Chem., 25, 11, 1635 (1953).

TABLE I

HEAT OF REACTION OF TEDCP WITH Na<sub>2</sub>S (4.184 joules = 1 calorie)

(1) Sample Weight, (gram)	(2) Volts	(3) Amps	(4) Time, (Seconds)	(5) Calibration, (joules/w)	(6) Average	(7) $\Delta T$ Sample, (w)	(8) $-\Delta H$ (kcal/mole)
0.20960	----- -----	----- -----	----- -----	0.04283 0.04289	0.04286	785.01	-20.62
0.38290	1.957956 1.956955 1.958957 1.939938	0.1216044 0.1216045 0.1217043 0.1206062	148.948 105.298 181.280 113.389	0.04319 0.04316 0.04301 0.04297	0.04308 .00005	1360.69	-19.67
0.20755	2.002000 1.98198 1.978977 1.975974	0.1228000 0.121602 0.1216023 0.1210026	78.299 174.693 93.335 174.439	0.04327 0.04300 0.04288 0.04271	0.04296 .00012	706.68	-18.80
0.20393	2.213211 2.215213	0.1377789 0.1378787	82.937 95.894	0.04380 0.04338	0.04359	796.66	-21.88
0.20020	2.197195 2.199197 2.197195 2.197195 2.195193 2.1986965 2.193191	0.1367805 0.1369103 0.1367805 0.1367805 0.1366807 0.13688035 0.1365809	98.871 102.335 156.203 255.825 151.839 137.333 122.420	0.04238 0.04252 0.04262 0.04225 0.04239 0.04290 0.04267	0.04253 .00008	736.97	-20.12

TABLE I (CONT):

(1) Sample Weight, (gram)	(2) Volts	(3) Amps	(4) Time, (Seconds)	(5) Calibration, (Joules/uv)	(6) Average	(7) $\Delta^T$ Sample, (uv)	(8) $-\Delta^T$ Hr kcal/mole
0.20069	2.226224	0.1368076	95.714	0.04173	0.04176 $\pm$ .00006	773.80	-20.69
	2.219217	0.1363783	83.873	0.04164			
	2.228226	0.1365774	86.214	0.04199			
	2.223221	0.1362779	94.871	0.04170			
	2.231229	0.1367771	140.747	0.04172			
0.20086	2.165163	0.1347837	117.721	0.04239	0.04235 $\pm$ .00005	751.27	-20.35
	2.176174	0.1354826	101.771	0.04241			
	2.181179	0.1358829	63.217	0.04221			
	2.175173	0.1353827	64.794	0.04237			
0.20051	2.228226	0.1354774	86.217	0.04239	0.04212 $\pm$ .00011	732.86	-19.78
	2.216214	0.1346786	139.548	0.04174			
	2.223221	0.1356779	73.030	0.04200			
	2.218216	0.1353784	77.722	0.04203			
	2.181179	0.1358821	96.914	0.04210			
	2.257255	0.1350745	66.028	0.04255			
0.20005	2.185183	0.1359817	99.263	0.04200	0.04184 $\pm$ .00008	677.64	-18.21
	2.185183	0.1351817	124.052	0.04157			
	2.183181	0.1350879	84.015	0.04185			
	2.187185	0.1352815	180.875	0.04180			
	2.187185	0.1361815	100.995	0.04202			



TABLE I (CONT):

(1) Sample Weight, (gram)	(2) Volts	(3) Amps	(4) Time, (Seconds)	(5) Calibration, (joules/uv)	(6) Average	(7) $\Delta T$ Sample (uv)	(8) $-\Delta_{Hr}$ kcal/mole
0.19957	2.206204	1.1354796	78.815	0.03541			
	2.240238	0.1353762	62.653	0.03510			
	2.230228	0.1357772	94.026	0.03531			
	2.219217	0.1355783	102.318	0.03532			
	2.2187165	0.1355784	184.576	0.03532			
	2.220218	0.1357282	303.685	0.03554	0.03534 $\pm$ .00006	810.34	-18.44
0.20035	2.182180	0.135782	72.158	0.03617			
	2.181179	0.1356821	62.969	0.03612			
	2.176174	0.1353826	125.692	0.03584			
	2.183181	0.1357819	64.128	0.03579	0.03598 $\pm$ .00010	792.33	-18.28
0.20002	2.160158	0.1343842	106.335	0.04216			
	2.163161	0.1345839	153.54	0.04221			
	2.164162	0.1345838	142.995	0.04195	0.04210	738.36	-19.97
0.19970	2.211209	0.1338791	79.065	0.04226			
	2.239237	0.134463	60.266	0.04246			
	2.207205	0.1345795	108.222	0.04243			
	2.206204	0.1345796	214.632	0.04214			
	2.208206	0.1346794	124.59	0.04193	0.04224 $\pm$ .00010	744.88	-20.25

Average  $\Delta_{Hr}$  = 19.8  $\pm$  0.3 kcal/mole

TABLE II  
ANALYSIS FOR  $\text{ClO}_4$  AFTER COBALT IS PRECIPITATED

<u>Sample Weight, (mg.)</u>	<u>Calculated Weight, (<math>\text{ClO}_4</math>, mg.)</u>	<u>Actual Weight, (<math>\text{ClO}_4</math>, mg.)</u>	<u>Difference, (mg.)</u>
500.2	277.6	277.7	✓ 0.1
502.3	278.8	277.2	- 1.6
502.3	278.8	277.7	- 1.1
503.0	279.1	278.3	- 0.8
503.8	279.6	277.6	- 2.0
504.1	279.8	277.4	- 2.4
Average		<u>278.783</u>	
		277.65	

TABLE III

ANALYSIS FOR  $\text{Co}_2\text{S}_3$ 

<u>TEDCP Weight,</u> <u>(mg.)</u>	<u><math>\text{BaSO}_4</math>, Found,</u> <u>(mg.)</u>	<u>Calculated</u> <u><math>\text{Co}_2\text{S}_3</math>, (mg.)</u>	<u>Actual <math>\text{Co}_2\text{S}_3</math>,</u> <u>(mg.)</u>	<u>Difference,</u> <u>(mg.)</u>
500.2	326.7	99.58	99.87	✓ 0.29
500.4	344.4	99.65	105.28	✓ 5.63
504.3	329.8	100.40	100.82	✓ 0.42
500.7	341.7	99.68	104.46	✓ 4.78
502.3	365.6	100.00	111.76	✓ 11.76
503.0	359.0	100.14	109.75	✓ 9.61
505.1	360.8	100.56	110.30	✓ 10.26
503.5	346.4	100.24	105.90	✓ 5.66
503.4	377.7	100.22	115.46	✓ 15.24
504.5	373.0	100.44	114.03	✓ 13.59

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 (U) The heat of formation ( $\Delta H_f^\circ$ ) of Triethylenediamine Cobalt III Perchlorate (TEDCP) was calculated from the reaction:  

$$2 \text{ Co}(\text{NH}_2(\text{CH}_2)_2\text{NH}_2)_3 (\text{C}_{10}\text{H}_{13})_3 + 3\text{Na}_2\text{S} \rightarrow \text{Co}_2\text{S}_3$$

$$6\text{NaClO}_4 + 6 (\text{CH}_2\text{NH}_2)_2$$
 and a value of -196 kcal/mole was obtained. The heat evolved from the above reaction was determined in a differential calorimeter.

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 I. Lenchitz, Charles  
 II. TEDCP  
 III. Title  
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